Shrinkage and Drying-out of Cement.
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THE following report of research work undertaken by the Concrete Roads Committee of the German Society for Research on Roads on the shrinkage and evaporation of water from cement during the drying period, in order to obtain a quick and accurate method of estimating the shrinkage of a given cement, is translated from "Zement."

The Present Method of Measuring Shrinkage.

The specimens for measuring shrinkage are three prisms made of plastic mortar measuring 4 cm. by 4 cm. by 16 cm. (1·6 in. by 1·6 in. by 6.3 in.) prepared in the same way as prisms for testing the strength in bending, except that they are kept in the moulds for two days instead of twenty hours in order to diminish the risk of the studs on the ends being broken off. After a storage of five days in water the weight and length of the specimens are determined by the Graf-Kaufmann apparatus. The prisms are then stored in an airtight box above a saturated solution of potash containing a liberal proportion of undissolved potash (formerly the prisms were stored over sulphuric acid of a density of 1·331). A saturated solution of potash when in equilibrium produces an atmosphere of 47 per cent. relative humidity; sulphuric acid of a density of 1·331 produces an atmosphere of 50 per cent. relative humidity. At the age of 28, 56, 90, and 180 days respectively the specimens are tested with regard to alteration in length and loss of weight. An iron prism is used as standard length, and its change of length is taken into account as a correction. The average of the results obtained from the three prisms is considered to be the correct value.

By taking the average, errors due to inaccuracy in determining the lengths and weights of the three prisms are minimised, as well as variations in the specimens with regard to shrinkage and evaporation. Errors which equally affect all three specimens are, however, not corrected. These are: (1) inaccuracy in making the prisms; (2) inaccuracy in measurements; (3) differences in storage

The first two possibilities of error partly produce accidental errors which would be eliminated if the tests were repeated many times. This will be referred to later. The method of storage in the box, however, produces systematic errors in the result, as will be shown. The amount of shrinkage and evaporation depends on the humidity of the atmosphere in the box which is again determined by two factors, namely, the partial pressure of the vapour in equilibrium above the drying medium and the speed at which the evaporated water is absorbed.

The present method of measurement takes into account only the first point in that it prescribes a drying medium which produces a given vapour-tension. The drying medium previously prescribed (diluted sulphuric acid of a density of 1·331) also had the disadvantage of further dilution when taking up water and so increasing the vapour-tension. This error is avoided if, as is now usual, a saturated solution of potash with undissolved potash is used, since when water is taken up the consistency of the solution remains the same.

Much more important is the fact that the actual partial pressure of the vapour in the box exceeds the theoretical pressure considerably during the first weeks. *Table* 1 shows the result of a test during which the humidity inside the box was measured directly at intervals by a suitable hygrometer the sensitive part of which could be inserted into the box like a thermometer.

TABLE 1.

ACTUAL HUMIDITY IN THE BOX WHEN THE SPECIMENS ARE
STORED TOGETHER.

Age.	Relative humidity above sulphuric acid.	Relative humidity above potash.
7 days	85·0 per cent.	82·0 per cent.
8 ,,	89·0 ,,	87·5 ,,
9 ,,	87·0 ,,	85·5 ,,
10 ,,	83·0 ,,	82·0 ,,
12 ,,	75·0 ,,	74·0 ,,
17 ,,	72·0 ,,	69·0 ,,
21 ,,	67·5 ,,	62·0 ,,
28 ,,	65·0 ,,	61·5 ,,

It will be seen that the values considerably exceed the theoretical limits of 50 and 47 per cent. relative humidity respectively. After eight days (i.e., after one day of drying out) the maxima of 89 and 87·5 per cent. respectively were recorded, and after 28 days the values were as high as 65 and 61·5 per cent. The drying process in the box therefore takes place in an atmosphere of high humidity at the beginning which gradually decreases.

It follows that the conditions in the box do not in any way resemble practical conditions, as the following consideration will show.

Consider two cements differing very much in respect of evaporation under the same conditions of drying out and which are used in concrete laid in two adjoining portions of a road, and of which test pieces are also made for testing shrinkage. If the cement in the road is allowed to dry out the relative humidity

will be practically the same in both portions owing to the convection of the atmosphere, whereas in the box the specimens with great evaporation will produce an atmosphere of greater humidity and will therefore dry out comparatively more slowly than the specimens with small evaporation. The cement with great evaporation, therefore, appears to have a comparatively smaller shrinkage.

Influence of Different Kinds of Storage.

The first problem was, therefore, to determine the influence which various different kinds of storage have on shrinkage and evaporation, and to find which part of the drying process slowly produces an equilibrium. The processes are,
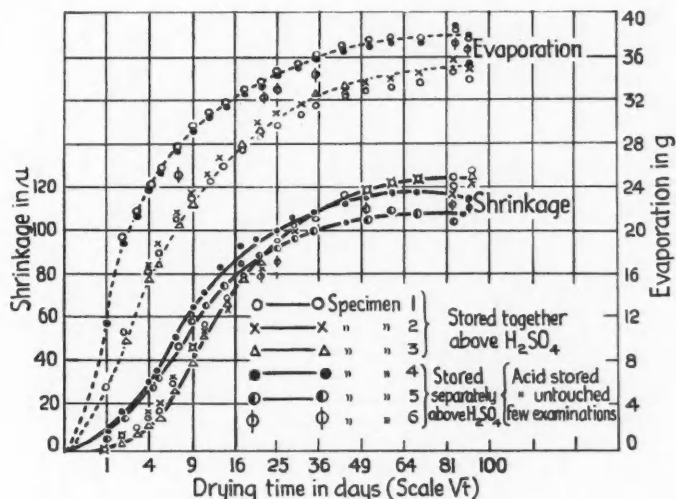

Fig. 1.

in sequence: (1) diffusion from the inside of the specimens to the surface; (2) evaporation from the surface of the specimens to the atmosphere; (3) diffusion through the atmosphere to the surface of the drying medium; (4) condensation at the surface of the drying medium; (5) diffusion from the surface to the inside of the drying medium. Processes (3) to (5) are the possible causes which slow down the production of equilibrium.

For purposes of investigation two series of prisms were made of the same Portland cement. Specimens (1) to (3) (Series 1) were all stored together in the usual way in one box above sulphuric acid but were examined at short intervals (at the beginning daily). The acid was frequently renewed so that its density could only change very little. The prisms of the second series were stored

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separately in boxes. Specimens (4) and (5) were examined at the same times as specimens (1) to (3). The acid under specimen (4) was stirred at each examination, the acid under specimen (5) was, however, left untouched. Specimen (6) was only examined at long intervals (at ages of 14 and 28 days and later periods). The humidity of this box was measured with a hygrometer as described. The results are shown in *Table* 2.

TABLE 2.

RESULTS AFTER DIFFERENT KINDS OF STORAGE.

Drying out days.	Age (days).	1 to 3 (Average).		4		5		6		Relative humidity. Per cent.
		S μ	E g	S μ	E g	S μ	E g	S μ	E g	
0	7	0	0·0	0	0·0	0	0·0	0	0·0	73
1	8	1	5·4	6	11·2	4	11·2	—	—	70
2	9	6	10·6	15	18·8	12	19·0	—	—	67
3	10	11	13·9	23	21·3	20	21·6	—	—	65
4	11	14	16·3	29	23·9	27	23·8	—	—	63
5	12	18	18·0	35	25·5	32	25·5	—	—	61
7	14	29	21·1	49	27·7	47	27·6	30	25·1	58
9	16	43	23·5	63	29·4	59	29·5	—	—	55
11	18	54	25·0	71	30·6	66	30·5	—	—	55
14	21	66	26·3	83	31·6	76	31·4	—	—	55
17	24	78	28·0	92	32·6	85	32·6	—	—	55
21	28	86	29·4	96	33·5	89	33·7	80	32·4	55
25	32	94	30·7	100	34·5	93	33·7	87	33·0	—
30	37	103	31·6	106	35·2	97	35·4	—	—	—
35	42	106	32·2	108	35·8	99	36·0	99	34·5	—
42	49	113	33·1	111	36·5	102	36·6	—	—	—
49	56	117	33·8	115	37·0	105	37·1	109	36·9	—
58	65	120	34·2	118	37·3	108	37·2	—	—	—
70	77	122	34·6	118	37·6	107	37·4	—	—	—
84	91	121	35·6	114	38·4	105	38·3	114	37·2	—
91	98	122	35·0	115	37·8	106	37·7	112	36·8	—

S = Shrinkage in μ = 0·001 millimetre.   E = Evaporation in grammes.

The process of drying out and shrinking is also illustrated graphically in *Fig.* 1. For reasons to be explained later the square root of the time has been used as abscissa. The process of evaporation produces, if represented like this, a curve which is strongly concave towards the axis of the abscissa (except for the first day). Evaporation is very great during the first days. After $1\frac{1}{2}$ days if stored separately, or five days if stored together, the specimens have given off one-half of the total amount to be given off.

The curves of shrinkage, however, are distinctly S-shaped. Shrinkage increases slowly during the first days and quicker later, both rates being referred to the square root of the time. In detail the results show the following.

(1) By storing the specimens separately a considerably more uniform percentage of humidity is obtained (compare *Tables* 1 and 2). At the beginning it is 73 per cent., decreasing after one day to 70 per cent. and after seven days to 58 per cent.

(2) The evaporation of one prism, if stored separately, amounts to 11·2 gr.

after one day ; the evaporation of the three prisms stored together totals 16·2 gr. This shows that if three specimens are stored together at least twice as much water can be given off as the acid is able to absorb in the same time.

(3) Not only at the beginning, but also at the end of the period, the amount of evaporation is greater when the specimens are stored separately than when they are stored together.

(4) The shrinkage when the specimens are stored together is considerably less at first than when stored separately owing to the smaller evaporation. But after three to four weeks the former reach the same values as the latter.

(5) The evaporations of specimen No. 4 (stirred acid) and specimen No. 5 (unstirred acid) are identical.   It follows that the increase of the speed of diffusion in specimen No. 4 does not produce greater evaporation.   Consequently the time of diffusion in the drying medium (process No. 5) can be neglected compared with the diffusion in the atmosphere of the box (process No. 3).

(6) The number of examinations also has an influence on the result. Evaporation is greater when the specimens are often examined (specimens Nos. 4 and 5) than when few examinations are made (specimen No. 6).

(7) The total shrinkage of specimen No. 4 exceeds, in spite of equal evaporation, that of specimen No. 5.

It follows from these results that storage in separate boxes approaches practical conditions much more closely than storage together.

Amount of Water Contained and Evaporated.

When it had been shown by these tests that storage in one box and storage in separate boxes produce different results, all further tests were made with specimens stored separately.   It was now necessary to determine how far shrinkage and evaporation depend on the amount of water contained in the specimens.

Plastic mortar is usually gauged with 15 per cent. of water (if the spread of the mortar is between 16 and 20 cm. [= 6 in. to 8 in.] after 15 " bumps " of the German standard apparatus).   After the mortar has been in the moulds for two hours it is smoothed off.   During this time some water has already been given off, and still more has evaporated by the time the mortar is taken out of the moulds at the end of two days.   During the storage in water up to the seventh day, however, water is again absorbed.   The amount of water contained at the beginning of the drying process on the seventh day cannot therefore be determined straight away.

It was now necessary to determine how the amount of water contained in the specimens alters in the various stages, and what connection it has with shrinkage and evaporation.   The tests were made as follows.   Two moulds were filled with mortar and the loss of weight after drying at a temperature of 110 deg. C. and ignition at a temperature of 700 deg. C. was determined.   After smoothing off (two hours after gauging) two samples were taken with a spoon from one of the three prisms and their loss of weight after drying and ignition

determined. After two and seven days small pieces were cut off with a chisel from the hardened prisms and immediately placed in a cupboard. The third prism, which remained intact, was stored separately in a box as described and its evaporation and shrinkage were determined as often as possible. These investigations were carried out in the same way with six different cements, i.e., two Portland cements, two iron Portland cements, and two Portland blastfurnace cements.

After four weeks the whole series was repeated. This time all investigations regarding the amount of water contained were made on the same prism so that two prisms remained for shrinkage tests. One of these was placed in a box after seven days, the second after fifty-six days.

Gauging water 15½ per cent., in all other cases 15 per cent.

1 ———— After gauging	4 ———— After 7 days
2 — — — — " smoothing off	5 —··—··— " 56 "
3 ············· " 2 days	6 —··—··— After 7 days
	7 —·—·—·— " 56 "
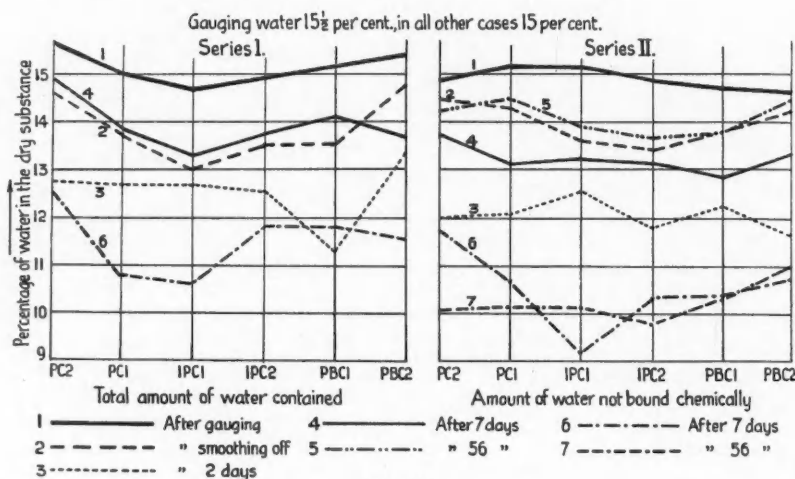
Fig. 2.

In the first series four of the six mortars were gauged with 15 per cent. of water, the two others, Portland cement No. 2 and Portland blastfurnace cement No. 2, with 15·5 per cent. (because with 15 per cent. gauging water the flow test gave too small a spread). In the second series all six mortars were gauged with 15 per cent. of water.
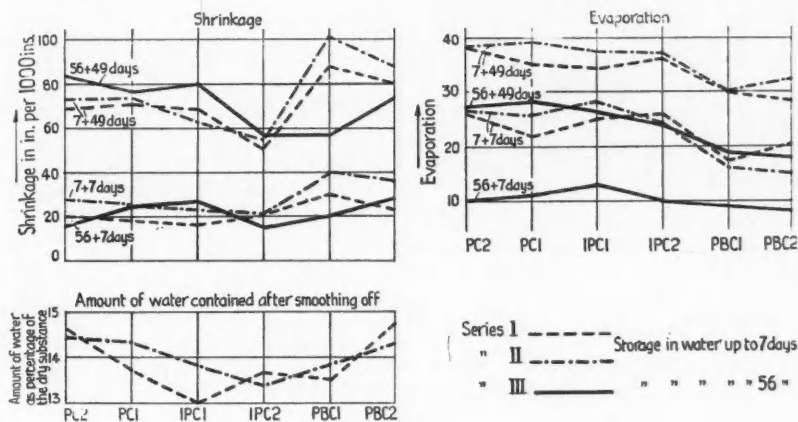
Fig. 2 shows the amount of water contained in the specimens. The values for the age of seven days, and especially those at two days, are not quite exact since much water is lost when the samples are cut off. The measurements on the samples differ by an average of 1 per cent. *Fig.* 1 shows the higher values, which for the reasons given may still be too low.

As can be seen from *Fig.* 2 a steady loss of water takes place between the time of gauging and taking the specimens from the moulds after two days, but this is compensated by storage in water approximately up to the amount contained when smoothed off after two hours. In the first series this stage is

reached after seven days ; in the second the seven-day values are considerably lower but the fifty-six day values are equal to those after two hours. Consequently the amount of water contained in the specimen after smoothing off (two hours after gauging) must be considered the most characteristic value for the true amount of water remaining in the mortar. This important value is shown in *Fig.* 3 in connection with the amount of water with which the specimens were gauged as well as with the values obtained for shrinkage and evaporation (only the fourteen and fifty-six day values are shown).

Fig. 3 illustrates the following.

(1) POSSIBILITY OF REPEATING THE TESTS.—The true amount of gauging water differs in the first series from the required value by an average of o·1 per cent. and in the second series by an average of o·2 per cent. The maximum

Fig. 3.

deviation in each series is o·4 per cent. After the smoothing off of the test-pieces the amount of water contained in the second series exceeds that of the first series by an average of o·17 per cent. Taking into account, however, that the actual amount of gauging water in the second series was lower by an average of o·28 per cent., the average increase becomes o·45 per cent. This difference is probably caused by the difference of the atmosphere on the days of gauging. The values are as follows : + o·8 per cent., + o·5 per cent., + o·3 per cent., − o·2 per cent., + o·1 per cent., + o·5 per cent. The deviations are considerable, so that the amount of water contained in the finished mortar cannot be kept exactly the same when repeating tests.

(2) AMOUNT AND LOSS OF WATER.—Comparing the differences between the amount of water contained in the mortar in the first and second series with the differences in evaporation it is seen that the variation of these two values is rather similar. The amount of evaporation of a specimen during the drying

process depends, therefore, on the amount of water contained in it ; this, however, depends on incidental changes that cannot be controlled. The evaporation after storage in water up to fifty-six days is much slower than after storage after seven days, but the difference between the two series after forty-nine days of drying is constant, i.e. 11 ± 1 gr. Here again the evaporation depends on the amount of water contained in the prism when it is smoothed off. The effect of a different amount of water on the shrinkage is, however, irregular. A definite relation cannot be established.

(3) AMOUNT OF WATER CONTAINED AND WORKABILITY.—Lastly, a definite relation between the amount of water remaining in the mortar and its workability was looked for, but the difficulty of repeating the tests accurately does not allow a precise statement to be made.

The workability of the six cements was measured by determining the viscosity of cement paste gauged with various amounts of water. The sequence was the same as that of the amount of gauging water to obtain a normal consistency when tested with the Vicat apparatus. In *Fig.* 3 the cements are shown on the abscissa according to the amount of gauging water in increasing order (= decreasing workability*). The two Portland cements need the smallest amount of water, and the two Portland blastfurnace cements need the greatest amount. There is an approximate relation between the workability and the amount of water contained after smoothing off Portland and iron Portland cements in such a way that with a larger amount of gauging water (= smaller workability) the amount of water contained after smoothing off will be smaller. This does not apply to Portland blastfurnace cements which show deviations in other respects also.

Shrinkage and Evaporation.

On the two series of specimens, three series of shrinkage and drying tests have been made as already mentioned, namely : (1) Series I, storage in water up to seven days ; (2) Series II, storage in water up to seven days ; (3) Series III, storage in water up to fifty-six days. In each series only one prism was available for each cement and this was stored separately in a box above potash. Examinations were made as often as possible, i.e., at the beginning daily and later at longer intervals. The results are shown in *Figs.* 4 to 9. In each figure the results of all three series of tests on one cement are shown. The results will be discussed from various points of view.

(1) SHAPE OF THE CURVES.—The same scale has been used for the co-ordinates in *Figs.* 4 to 9 as in *Fig.* 1. The square root of the time has been used as abscissa. Consider first the curves marked 1*a*, 1*b*, 2*a*, 2*b* representing shrinkage and evaporation after storage in water up to seven days. The shape of the curves for Portland cements (*Figs.* 4 and 5) and iron Portland cements (*Figs.* 6

* The term " workability " is related to the properties of the dry cement. The test pieces were all of the same consistency, but different quantities of water were needed with different cements. Cements which needed more water to reach this consistency are described as cements of " low workability."
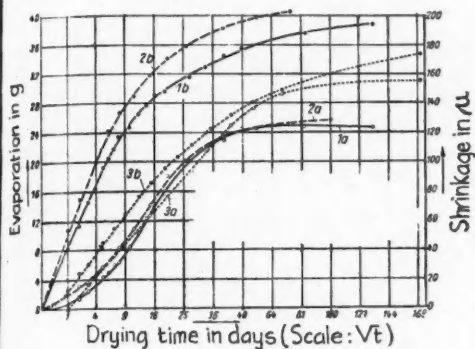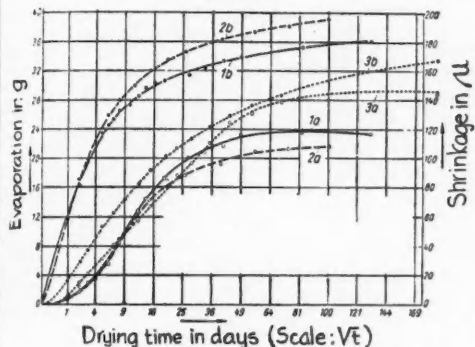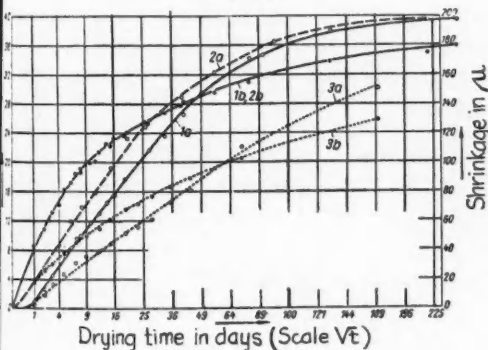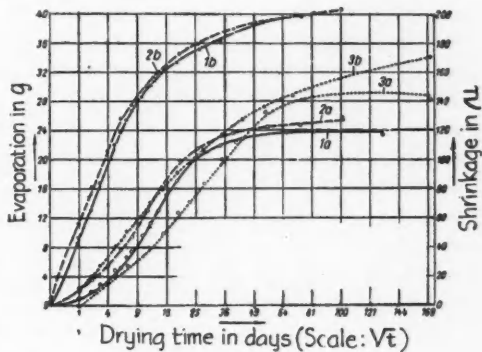
Fig. 4.

Fig. 5.

Fig. 6.

Fig. 7.

Fig. 8.
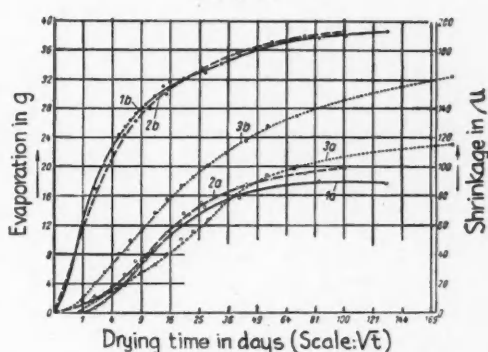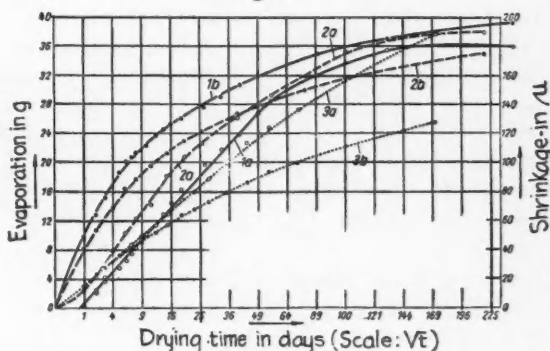
Fig. 9.

SHRINKAGE	EVAPORATION	
1a ○———○	1b ●———●	Series I 7 days in water
2a ○— —○	2b ●— —●	" II 7 " " "
3a ○········○	3b ●········●	" III 56 " " "

and 7) is the same as in *Fig.* 1. The curve of evaporation rises almost in a straight line during the first days and then bends sharply towards the axis of the abscissa. The curves of shrinkage are S-shaped, as in *Fig.* 1. After about seventy days of drying out the process of shrinkage is practically completed. The evaporation, however, continues to increase slowly.

The corresponding curves for Portland blastfurnace cements are distinctly different. The process of evaporation is much slower than in other cements. The curve of shrinkage rises from the second day of drying for about four weeks almost in a straight line and then bends slowly downwards. The process is only completed after about 200 days. The chief difference compared with other cements is this linear form of the shrinkage curve corresponding to an increase proportional to the square root of time.

The drying out after water storage up to an age of fifty-six days, however, gives a totally different picture. The process of drying out is even slower than in Portland blastfurnace cement after storage up to seven days. The shape of the curve is the same for all kinds of cement, i.e., as has just been described for Portland blastfurnace cements, the curve of evaporation is slightly curved towards the abscissa and the curve of shrinkage is almost linear during a period of about seven weeks. It is remarkable that only the evaporation is considerably smaller than after storage up to seven days; the shrinkage is generally of the same magnitude, and after some time reaches even higher values.

(2) POSSIBILITY OF REPETITION.—The curves of evaporation obtained from series 1 and 2 partly agree and partly show considerable differences. As already mentioned, the differences in evaporation are due to the amount of water contained in the mortar after smoothing off. It can be assumed that the process of evaporation could be repeated if the amount of water contained in the mortar after smoothing off could be kept the same.

The curves of shrinkage agree except for their origin. The curves for Portland blastfurnace cements and for storage up to fifty-six days are linear and do not pass through the origin. Consequently the beginning of the process is far less accurately determined than the other points. This is natural since the temperature of the specimen taken out of the water decreases rapidly owing to sudden evaporation which causes a change of length that cannot be controlled. The value after seven days must therefore be rejected as an origin. The value after eight days (= one day of drying) is more suitable. Owing to the great number of examinations it is possible to estimate the deviation from the true position of the curve. The deviations extend to about $\pm 3 \mu$. If in particular cases the deviation is higher this is due to an error in the adjustment of the reference standard bar. These deviations can only be noticed because the values for a series of different cements on one day are all on the same side of the curves. For instance, in the second series of tests (*2a* in *Figs.* 4 to 9) the values for the shrinkage after twenty days of drying out are all too high by 4μ.

Swelling and Absorption.

In order to complete the investigation two specimens of the same cement (Nos. 2 and 5 in *Table 2*) were used to investigate the absorption of moisture and the corresponding increase in length when stored above water after a drying period of ninety-one days. The results of this series were plotted. The scales are the same as before. The process is even slower than the drying out after storage in water up to fifty-six days. The curve of the amount of water absorbed is almost linear, and therefore the amount is proportional to the square root of the time. The curve of swelling is, however, sharply curved towards the abscissa from the beginning. The curve of swelling is therefore more convex than the curve of the amount of water absorbed, whereas the opposite relation exists between shrinkage and evaporation.

Theory of the Process of Drying Out.

It is impossible to distinguish between the two phases of the drying process : evaporation of the water in the pores, and evaporation of water induced by capillary attraction. It was attempted to calculate the process of evaporation by the simple Fick law of diffusion. A simple mathematical solution is possible only for one theoretical case, namely, one surface bounding a body of infinite extension. The amount of water (q) diffused during the time (t) through the surface is given by the formula

$$q = k\sqrt{t}.$$

Owing to this relation the square root of the time has been used as the abscissa in all the diagrams. The figures show that the $\sqrt{t}$-relation is more correct in most cases than one would expect with such a rough approximation. In any case the $\sqrt{t}$-scale is the adequate graphic representation of the drying process.

Conclusions Regarding the Determination of Shrinkage and Evaporation.

(1) The evaporation is so great during the first days that the amount of water given off by three specimens simultaneously cannot be absorbed by the drying medium. Consequently the drying process is disturbed. It is therefore convenient to store the specimens separately in boxes and only put them together when most of the water has been evaporated (after about seven days).

(2) Most of the drying and shrinking takes place during the first weeks. It is not convenient, therefore, to make the first measurement after three weeks (at the age of twenty-six days). The increase of shrinkage after this time is only 30 to 45 per cent. of the amount reached after twenty-eight days in Portland and iron Portland cements (see *Table 5*). It is convenient to make the measurements at such intervals that, represented at a scale of $\sqrt{t}$, the intervals are equal. This condition is fulfilled exactly by the series of squares 0, 1, 4, 9, 16, etc., and also approximately by the series 0, 1, 3, 7, 13, 21, 31, 43, 57, 73, etc. This corresponds to the ages of 7, 8, 10, 14, 20, 28, 38, 50, 64, 80, etc., days. This series is convenient because it contains the fourteen-day and twenty-eight-day

values. Measurement after twenty-eight days is not necessary for determining shrinkage. The values for fifty and eighty days can be taken for checking.

(3) The determination of the zero point of shrinkage is uncertain owing to changes in temperature caused by sudden evaporation which cannot be controlled. It is therefore advisable to take the value for one day of drying as zero point of shrinkage. *Tables* 3a and 4a show how far the results of series 1 and 2 agree in spite of apparent differences.

TABLE 3a.

ZERO POINT SEVEN DAYS. SHRINKAGE IN μ.

Age in days.	PC1			PC2			IPC1			IPC2			PBC1			PBC2		
	1	2	Δ	1	2	Δ	1	2	Δ	1	2	Δ	1	2	Δ	1	2	Δ
7	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
8	3	10	+7	5	10	+ 5	5	5	0	0	6	+6	4	17	+13	2	13	+11
10	12	19	+7	14	23	+ 9	14	14	0	8	13	+5	22	38	+16	19	31	+12
14	30	37	+7	33	44	+11	37	36	− 1	25	28	+3	46	62	+16	38	56	+18
20	56	63	+7	62	76	+14	70	65	− 5	45	49	+4	69	87	+18	59	82	+23
28	86	90	+4	91	100	+ 9	90	83	− 7	62	67	+5	93	112	+19	84	104	+20
38	106	108	+2	107	113	+ 6	104	93	−11	74	79	+5	117	133	+16	102	121	+19
50	118	119	+1	114	120	+ 6	112	98	−14	82	87	+5	138	149	+11	124	135	+11
80	124	125	+1	119	124	+ 5	118	106	−12	89	95	+6	167	174	+ 7	155	158	+ 3
$\sqrt{\dfrac{\Sigma\Delta^2}{7}}$			5.7			9.3			8.7			5.3			16.2			16.9

TABLE 3b.

ZERO POINT SEVEN DAYS. SHRINKAGE IN IN. PER 1,000 IN.

Age in days.	PC1		PC2		IPC1		IPC2		PBC1		PBC2	
	1	2	1	2	1	2	1	2	1	2	1	2
7	—	—	—	—	—	—	—	—	—	—	—	—
8	0.02	0.06	0.03	0.06	0.03	0.03	0.00	0.04	0.03	0.11	0.01	0.08
10	0.08	0.12	0.09	0.14	0.09	0.09	0.05	0.08	0.14	0.24	0.12	0.19
14	0.19	0.23	0.21	0.28	0.23	0.23	0.16	0.18	0.29	0.39	0.24	0.35
20	0.35	0.39	0.39	0.48	0.44	0.41	0.28	0.31	0.43	0.54	0.37	0.51
28	0.54	0.56	0.57	0.63	0.56	0.52	0.39	0.42	0.58	0.70	0.53	0.65
38	0.66	0.68	0.67	0.71	0.65	0.58	0.46	0.49	0.73	0.83	0.64	0.76
50	0.74	0.74	0.71	0.75	0.70	0.61	0.51	0.54	0.86	0.93	0.78	0.84
80	0.78	0.78	0.74	0.78	0.74	0.66	0.56	0.59	1.04	1.09	0.97	0.99
$\sqrt{\dfrac{\Sigma\Delta^2}{7}}$	0.036		0.058		0.054		0.033		0.101		0.106	

In *Table* 3a the results for the ages given (7, 8, 10, etc., days) are taken from *Figs.* 4 to 9. Besides the differences between single values the standard deviation also is given, calculated by the formula

$$\sqrt{\frac{\Sigma\Delta^2}{n-1}}$$

representing the inaccuracy when repeating the tests.

Table 3b corresponds to 3a except that the results are given in the usual form, i.e., in millimetres per metre. The table shows that the uncertainty in repeating the tests is as follows :

Portland cement ..	..	..	.. 0·036 and 0·058
Iron Portland cement	..	..	.. 0·033 ,, 0·054
Portland blastfurnace cement	..	..	0·101 ,, 0·106

If, however, the value for the eighth day is taken as zero point, as shown in *Tables* 4a and 4b, the standard deviation is much lower, as follows :

Portland cement ..	..	..	.. 0·026 and 0·030
Iron Portland cement	..	..	.. 0·011 ,, 0·059
Portland blastfurnace cement	..	..	0·031 ,, 0·057

TABLE 4a.

ZERO POINT EIGHT DAYS. SHRINKAGE IN μ.

Age in days.	PC1			PC2			IPC1			IPC2			PBC1			PBC2		
	1	2	Δ	1	2	Δ	1	2	Δ	1	2	Δ	1	2	Δ	1	2	Δ
8	—	—		—	—		—	—		—	—		—	—		—	—	
10	9	9	0	9	13	+ 4	9	9	0	8	7	—1	18	21	+ 3	17	18	+ 1
14	27	27	0	28	33	+ 5	32	31	— 1	25	22	—3	42	45	+ 3	36	43	+ 7
20	53	53	0	57	66	+ 9	65	60	— 5	45	43	—2	65	70	+ 5	57	69	+12
28	83	80	—3	86	90	+ 4	85	78	— 7	62	60	—2	89	95	+ 6	82	91	+ 9
38	103	98	—5	102	103	+ 1	99	88	—11	74	73	—1	113	116	+ 3	100	108	+ 8
50	115	109	—6	109	110	+ 1	107	93	—14	82	81	—1	134	132	— 2	122	122	0
80	121	115	—6	114	114	0	113	101	—12	89	89	0	163	157	— 6	153	145	— 8
$\sqrt{\dfrac{\Sigma\varDelta^2}{6}}$			4·2			4·8			9·5			1·8			4·9			9·1

TABLE 4b.

ZERO POINT EIGHT DAYS. SHRINKAGE IN IN. PER 1,000 IN.

Age in days.	PC1		PC2		IPC1		IPC2		PBC1		PBC2	
	1	2	1	2	1	2	1	2	1	2	1	2
8	—	—	—	—	—	—	—	—	—	—	—	—
10	0·06	0·06	0·06	0·08	0·06	0·06	0·05	0·04	0·11	0·13	0·11	0·11
14	0·17	0·17	0·18	0·21	0·20	0·19	0·16	0·14	0·26	0·28	0·22	0·27
20	0·33	0·33	0·37	0·41	0·41	0·37	0·28	0·27	0·41	0·44	0·36	0·43
28	0·52	0·50	0·54	0·56	0·53	0·49	0·39	0·38	0·56	0·59	0·51	0·57
38	0·64	0·61	0·64	0·64	0·62	0·55	0·46	0·46	0·71	0·73	0·62	0·68
50	0·72	0·68	0·68	0·69	0·67	0·58	0·51	0·51	0·84	0·83	0·76	0·76
80	0·76	0·72	0·71	0·71	0·71	0·63	0·56	0·56	1·02	0·98	0·96	0·91
$\sqrt{\dfrac{\Sigma\varDelta^2}{6}}$	0·026		0·030		0·059		0·011		0·031		0·057	

The comparison between *Tables* 3a and 3b, and 4a and 4b proves the superiority of the proposed method.

In practice it is important to get the shrinkage of a cement tested quickly. Therefore the figures in *Table* 4 have been transformed in such a way that the values for eight days were made zero and those for twenty-eight days 100 (*Table* 5).

TABLE 5.

ZERO POINT EIGHT DAYS. SHRINKAGE AS PERCENTAGE OF THE VALUES
FOR TWENTY-EIGHT DAYS.

Age in days.	PC1		PC2		IPC1		IPC2		PBC1		PBC2	
	1	2	1	2	1	2	1	2	1	2	1	2
8	—	—	—	—	—	—	—	—	—	—	—	—
10	12	12	10	14	11	12	13	12	20	22	21	20
14	33	34	33	37	38	40	40	37	47	47	44	47
20	63	66	66	73	76	77	73	72	73	74	70	76
28	100	100	100	100	100	100	100	100	100	100	100	100
38	123	122	119	115	116	113	119	122	127	122	122	119
50	139	136	127	122	126	119	132	135	151	139	149	134
80	146	144	133	127	133	130	143	148	183	165	187	160

The values for all other days are therefore percentages of the values for twenty-eight days. Shrinkage after fourteen days amounts to 30 to 40 per cent. of that after twenty-eight days. The Portland cements have the lowest, and the Portland blastfurnace cements the highest, percentage. Consequently if cements of similar quality are tested regularly it is possible to obtain reliable results regarding shrinkage after eight, ten, and fourteen days if the specimens are stored separately. The time needed for testing is thus reduced to about half the time prescribed in existing regulations. The later examinations (after twenty and twenty-eight days) only check the earlier tests. Testing at higher ages is unnecessary since the shape of the curve of shrinkage is known by experience. It is important to know that only in Portland blastfurnace cements does the shrinkage increase considerably at higher ages, whereas in Portland and iron Portland cements it exceeds the value for twenty-eight days only by 30 to 45 per cent.

Separation of Minerals in Cement Manufacture.

THE following notes describe the Breerwood process of separating minerals now used at several cement works in the United States, and are of considerable interest because they relate to the first plant specially designed to take full advantage of its possibilities and free from any limitation caused by existing equipment.

According to *Rock Products*, the entire raw material flow-sheet, from the quarry to the kiln-feed tanks, combines grinding and mineral-separating methods and equipment appropriate to the materials available at the site.   The principal results, and the conclusions that can be drawn from them after a year's working are as follows.

Cements of high quality and strength and of extraordinary uniformity have been produced by this process from compositions derived solely from a deposit which otherwise could not have been used.   The provisions for relatively simple adjustments in the operation of the plant make it possible to vary the ratios and moduli for the production of all known types of cement, including those which satisfy the most rigid specifications for sulphate-resisting cements.   The processing method reduces both soluble and insoluble alkalis, and the clinker is almost entirely free of alkali.   By recovering almost all the available lime and the useful proportions and varieties of clay minerals, the process assures a practically inexhaustible supply of raw material.   The combination of the grinding and mineral-separating methods has resulted in a corrected kiln feed averaging about 90 per cent. passing a 325-mesh, this high fineness resulting in a composition free from segregation and of excellent burning characteristics.   The high fineness is attained economically by eliminating the excessive proportion of coarse quartz prior to final grinding of the necessary minerals, thereby reducing the total power consumption of the two mills to a minimum.   The clinker is hard-burned but is easily ground, a characteristic at least partly attributable to the uniformity of the composition and its fineness.

Quarrying.

The entire raw material supply is adjacent to the plant and may be described as a consolidated marl, the two principal strata being quite hard, with the lower layer distinctly recrystallised.   The four major horizontal strata are (1) over-burden clay, (2) a soft lime-bearing material known locally as soft Tosca, (3) a somewhat harder stratum of similar material known as hard Tosca, and (4) a crystalline limestone known as Piedra.   The average analysis of the strata is as follows :

		Overburden Clay	Soft Tosca	Hard Tosca	Piedra
SiO_2..	..	68·08	66·30	19·48	9·68
R_2O_3	..	20·20	17·72	4·16	0·84
$CaCO_3$	..	2·70	5·17	71·90	89·30
$MgCO_3$	..	—	1·75	4·34	2·61
Ratio	..	3·31	3·74	4·68	11·52
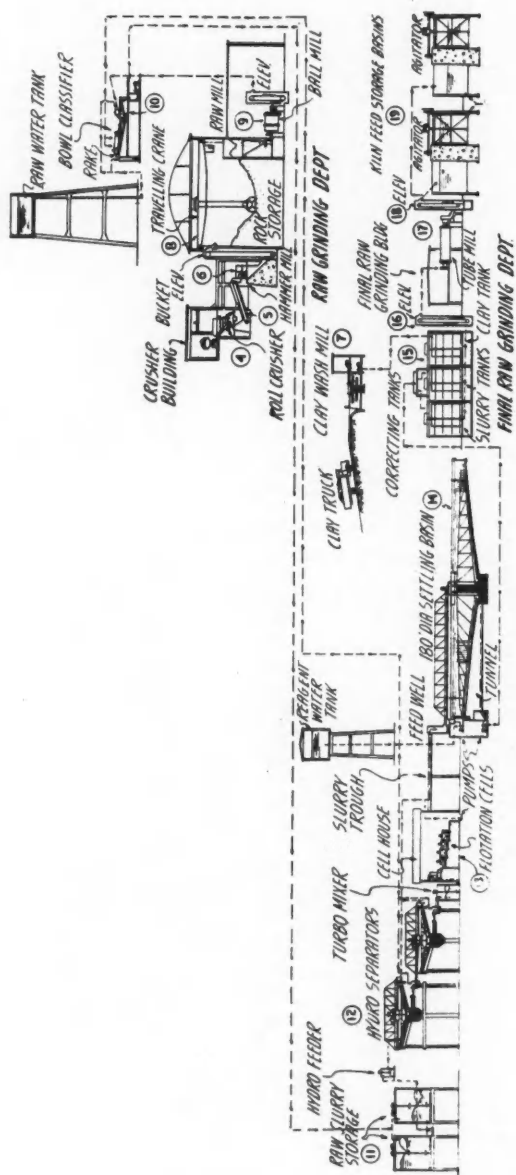
Fig. 1.—Flow Sheet from Crusher Building to Agitator.

The necessity for employing a mineral-separating method to correct the chemical composition of each of these strata or any combination of them is evident. The Piedra, or hard limestone, is higher in lime content than is required but it contains practically no iron and alumina. The silica content is misleading, as it occurs mostly as coarse quartz and is therefore undesirable as a constituent of the mixture ; the other lime stratum, the hard Tosca, is too low in lime and considerably excessive in silica, the SiO_2/R_2O_3 ratio being 4·68, a large proportion of the silica being in the form of coarse quartz grains. The soft Tosca, which is used as the source of clay minerals, must be corrected by subtracting a part of the silica, since its natural SiO_2/R_2O_3 ratio is 3·74. Even the overburden clay could readily be used if a small proportion of the silica were removed.

The present method of quarrying the material comprises stripping and discarding all overburden clay and about half of the soft Tosca, the remainder of the deposit being used. To simplify the operation of the process plant and to avoid frequent adjustment of the units, two cuts are made, the useful part of the soft Tosca, together with the hard Tosca, being sent to storage and kept in a pile separate from the Piedra. From these two piles, the materials are delivered by the crane to two bins above the ball mill and are proportioned by separate feeders in direct relation to the thickness of the strata, a relatively uniform material mixture being sent to the process plant. The average analysis of this is :

SiO_2	28·70
Fe_2O_3	2·57
Al_2O_3	4·01
CaO	33·35
$CaCO_3$	59·50
MgO	1·25
Loss	29·69
Total	99·55
SiO_2/R_2O_3	4·36

Petrographic Composition of the Materials.

Petrographic examinations of the materials, which comprised an essential part of the preliminary mineral-separation investigations, identified the principal component minerals as calcite, quartz, aluminum silicates, limonite, and dolomite. The aluminum silicates comprise the alkali feldspars, soda plagioclase (albite) and potash feldspar (microcline) and hydrous aluminum silicates of indeterminate composition approaching clay minerals and ranging in size from one to several microns. Accordingly, the mineralogical equivalent of the above analysis is approximately :

Quartz	24·40
Limonite	3·14
Aluminum silicates	9·78
Calcite	59·75
Dolomite	3·01
	100·08

A thorough preliminary investigation of these materials was made to determine whether they would respond satisfactorily to minerals separation methods, before the site was finally selected. The principal tests comprised determinations of grinding, classification and thickening characteristics, together with investigations of the amenability of each of the materials and their fractions, according to particle size and mixture, to froth-flotation concentration. The test results served as the basis in designing the flow diagram and the selection of the various units of the process plant.
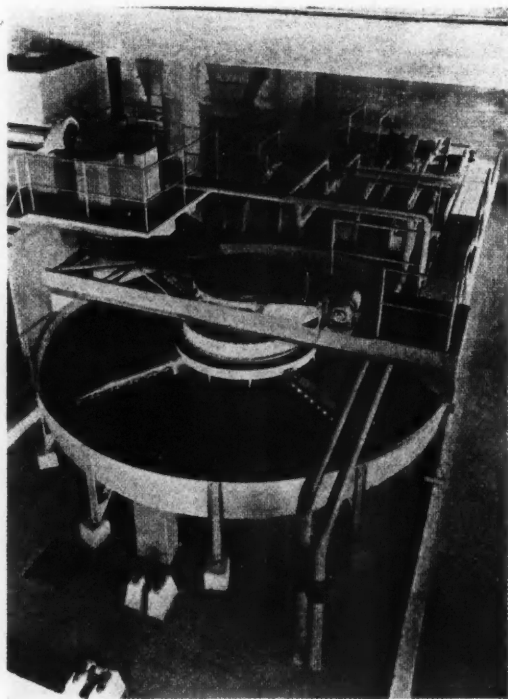
Fig. 2.—Bowl Classifier in the Raw-Mill Room.

In making the essential corrections of the materials, the principal objects of the process plant are (1) to recover substantially all the calcium carbonate and the useful fine clay minerals, (2) to subtract and discard the excessive proportion of silica in the form of coarse quartz grains without attempting to grind the latter, and (3) to control the proportion of alumina for the production of low-heat and sulphate-resisting cements.

Prior to storage, the materials are crushed to pass a $\frac{3}{4}$-in. ring in a roll and hammermill (*Fig.* 2), and stored in the two piles described. Preliminary grinding

of the proportioned materials is accomplished in a single 9-ft. by 11-ft. 6-in. ball mill in closed circuit with a bowl classifier (*Fig.* 2). This operation differs from the usual preliminary grinding, since its purpose is to reduce the materials only to a degree at which the coarse quartz is physically released, whereby it may be subtracted from the calcite and useful clay minerals in the flotation operation. Mineral bond breakage is complete when 85 per cent. is smaller than the 100-mesh sieve, further grinding at this point being undesirable as it would involve waste of power and increase the operating costs.

(To be Continued.)

Improvements in Cooling Clinker.

THE operation of a new type of cooler at the works of the Valley Forge Cement Co., Pennsylvania, U.S.A., is described in a recent issue of "Rock Products" by Mr. R. S. Torgerson. This cement mill works on the wet process, and it is stated that a considerable reduction in autoclave expansion and greater ease in grinding the clinker, due to the air quenching, have been obtained since the new cooler has been in operation. The low temperature of the clinker delivered to the mill has also increased the grinding efficiencies.

In 1927, two Polysius "Solo" kilns with attached coolers were placed in operation—the first installation of its kind in the United States. It is to these that improvements in the cooler have recently been made. The kilns had three
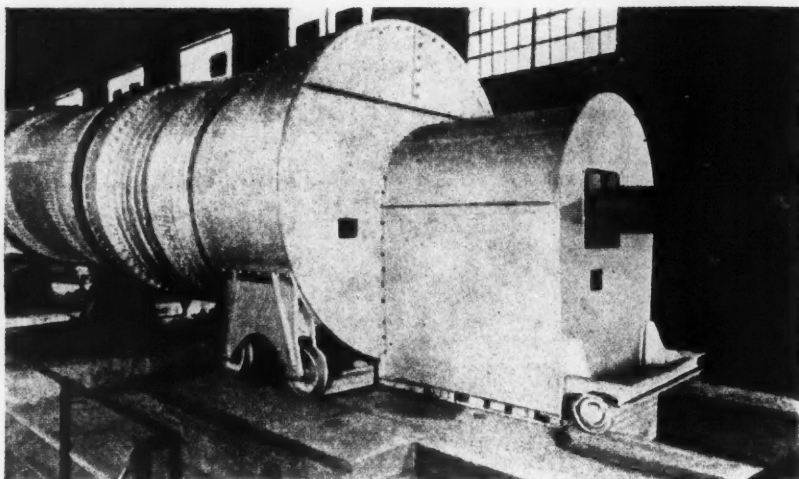
Fig. 1.—Preheated Air Chamber above Primary Clinker Cooling Unit on Kiln.

sections of different diameters; a double-shell cooling section, 9 ft. 10 in. in diameter and 31 ft. long; a burning section, 11 ft. 10 in. in diameter and 50 ft. long; and a preheating section, 8 ft. 10 in. in diameter and 142 ft. long. All the air for combustion was taken in through ports in the shell of the cooler section of the kiln. The kiln burners for pulverised coal, a special type made for the Solo kiln, consisted of a steel pipe which projected as a cantilever through the cooler section into the kilns, the length of the pipe being adjustable, so that the burning zone could be changed within limits, at will. The cooling section of the kiln discharged through air-locked, automatically controlled valves to "Ski-pulters," which extended from the ends of the cooler over a bridge structure to the west side of the storage building, where they discharged into the clinker storage through automatic scales.

The New Cooler.

With the installation of the new Fuller movable incline grate cooler, the cooler section of the old kilns became a part of the burning section. The illustrations (*Figs.* 1 to 3) show the firing end of the kilns as they now appear with the new cooler. A flow diagram (*Fig.* 4) of the installation shows the situation of the primary and secondary coolers. The primary unit is operated in accordance with the preheated air requirements of the kiln, which, in all cases, is sufficient for thorough quenching of the clinker. In general, the temperature of the clinker delivered to the primary unit averages approximately 2,500 deg. F., while that leaving the primary unit is between 800 deg. F. and 1,200 deg. F. As mentioned before, the primary unit is operated as an air quencher and an air preheater to the kiln. On the other hand, the secondary unit is operated as a

Fig. 2.—Interior of Primary Clinker Cooler.

final cooler, the speed of which is regulated to reduce the temperature of the clinker to between 120 deg. F. and 140 deg. F. when it is discharged to the clinker pit. The bed on the secondary unit is considerably thicker than on the primary unit and the cooler is operated at a slower speed.

It will be noticed from the illustrations that the cooler is of comparatively simple construction. The grates are of water-cooled cast-iron construction, and no high temperature metal is used in the construction of the cooler. The only moving parts are the rollers carrying the movable frame and the eccentric drive, all of which are situated outside the cooler housing.

The cooler can be built on almost any pitch up to 18 deg. without any clinker short-circuiting over the grate. It may also be built in any width and almost any length, with variable speed motor regulating the strokes of the movable grates and with the length of the strokes controlled on the eccentric.

The speed of flow over the grates can be varied in accordance with requirements.

As shown in the illustration, the stationary grates are mounted on the wall frames (indicated by shading), and the movable grates are supported on the movable frame and carried by the four rollers. There are air holes or slots through both the secondary and stationary grates. This is one source of quenching air which is fed directly up through the bed of the clinker. Another supply of air enters between the movable and stationary grates where the clinker drops from grate to grate on both the back and forward strokes. The water-cooling system is relatively simple, as the movable grates are on one complete circuit and the stationary grates on another complete circuit. The only flexible connections are at the top and bottom of the grates. The water connections through the entire length of the grates are of rigid construction.
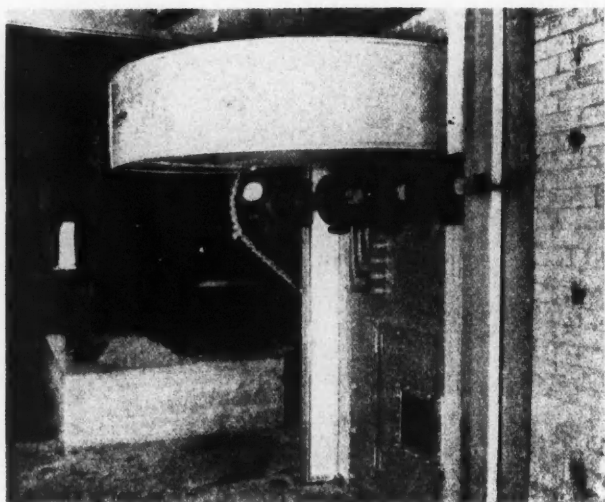
Fig. 3.—Air Fan, Air Duct, and Eccentric Drive of Cooler.

Single-Stage Cooling.

When final and complete cooling is desired on a single-stage unit, the cooler is built with a divided air box and a swinging gate placed near the middle of the grate above the clinker bed. In this case the preheated air from the upper half of the grate is delivered to the kiln, while the air from the lower half is delivered to the atmosphere. A single unit of this construction accomplishes the same results as the two-stage units at the Valley Forge plant. However, the use of separate primary and secondary units provides a slightly better control of the discharge temperature of the clinker than is possible with one, as it is possible to regulate the thickness of the bed and the speed of the secondary unit to obtain final cooling
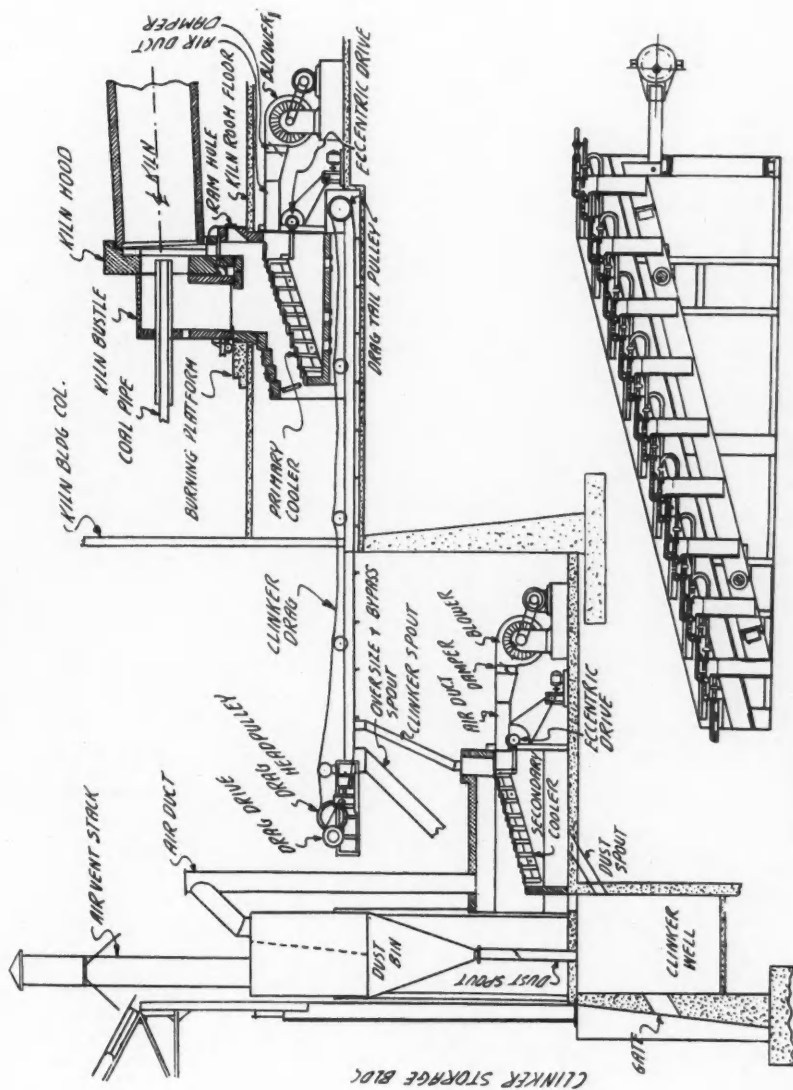
Fig. 4.—Above : Flow Diagram of Clinker Cooler Units.
Below : View of Clinker Cooler Grates.

irrespective of the temperature of the material delivered to it by the primary or quenching unit.

A continuous single grate is now being designed by the Fuller Co. in which the upper half of the grates can be operated at one speed, while the lower half can be operated at a slower speed or shorter stroke, and it is hoped with this design to have the same flexibility as is possible with two separate units.

Recent Patents Relating to Cement and Lime.

Aluminous Cement.

490,421. Calloy, Ltd., and G. N. Kirsebom. February 11, 1937.

In a method of preparing alloys of aluminium and calcium in which an oxygen-containing compound, other than a sulphate or oxyhalide, of calcium is heated in a bath of molten aluminium, the slag which is formed in addition to the alloy may be used for the preparation of cements.

Refractory Substances.
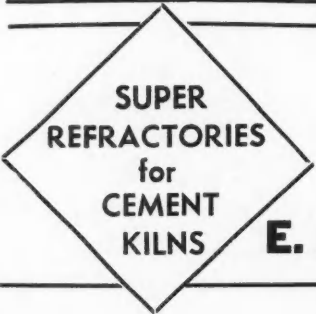
489,680. R. P. Heuer. August 3, 1937.

The bricks for a suspension type furnace roof are made of a refractory consisting mainly of chrome ore with magnesia as the chief minor constituent, the ore being coarsely ground to particles, ranging in screen size between 3 and 20 mesh per linear inch, while the magnesia is more finely ground, e.g. to pass at least a 48-mesh screen. The grading into coarse and fine particles as between the ore and magnesia may vary, but preferably more than 80 per cent. of the particles larger than 10-mesh size are ore, and more than 80 per cent. of the finer particles are magnesia. When the bricks are to be burnt in situ a bonding agent is used, e.g. sulphuric acid, acid sodium sulphite, waste sulphite liquor or dextrin, and the bricks are dried at a temperature between 212 and 572 deg. F.

487,213. E. J. Lavino & Co., January 21, 1938.

A magnesia refractory comprising grains of crystalline magnesia coated and cemented together by a material consisting chiefly of forsterite is formed by firing a mixture of coarse grain of sintered magnesite and finely-divided silica in such proportions that the silica can combine with part of the magnesia to form forsterite. In an example, 100 parts of deadburnt crystalline magnesia crushed to pass a 6-mesh screen but with 50 per cent. retained on a 100-mesh screen (U.S.A. Bureau of Standards) and 14 parts of silica flour are dry mixed, tempered in a wet pan, moulded, and fired between 3100 and 3450 deg. F.
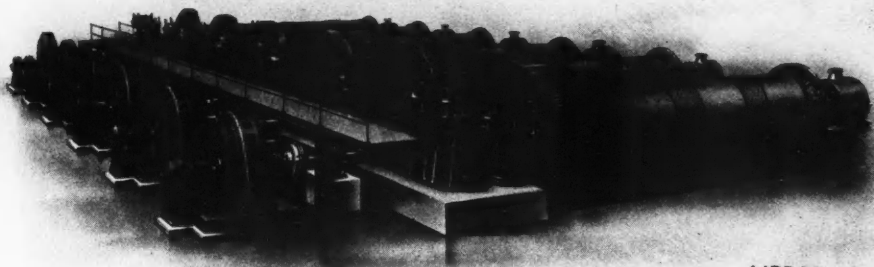
We illustrate above a group of tube mills erected by us in a large cement works

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